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TNO report

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An automated air sampling system comprising an OPTIC GC injector I - Quantification of soman vapour

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Een geautomatiseerd luchtmonstername-systeem met een OPTIC gaschromatografie-injector I. Concentratiebepaling van uitstromende soman-damp

Probleemstelling

In het kader van het project Detectie-apparatuur chemische strijdmiddelen beschikt TNO Defensie en Veiligheid, locatie Rijswijk sinds jaar en dag over een zogenaamde dampgeneratieopstelling. Met deze opstelling kunnen dampen met bekende concentratie worden gegenereerd van alle bekende strijdmiddelen. In het ideale geval is de concentratie van het strijdgas in het uitstromende gas te allen tijde bekend (in 'real time'). Hiertoe werd in het verleden een Meloy-vlamfotometer ingezet die op fosfor danwel zwavel analyseert. In de loop van de tijd ontstond de behoefte om het uitstromende gas ook op samenstelling te analyseren zodat meer zekerheid bestaat over de aard van de verbinding en eventuele verontreinigingen c.q. afbraakproducten.

Daarvoor werd een gaschromatograaf met een ingebouwde thermodesorptie-injector aangeschaft. Na enkele jaren werd de vaste thermodesorptie-injector vervangen door een meer flexibel thermodesorptiesysteem dat onder meer bestaat uit een OPTIC GC-injector. Deze optie biedt de mogelijkheid tot automatiseren van de verificatie van de dampconcentratie. Dit rapport beschrijft de werking en eerste validerende tests met dit systeem en voorziet hiermee in deliverable 305.

Beschrijving van de werkzaamheden

Het OPTIC-systeem werd getest met het zenuwgas soman. Verschillende concentraties werden gemaakt zowel in droge als in zeer vochtige lucht. De resultaten werden vergeleken met een 'solvent desorption'-methode die de referentie is.

Resultaten en conclusies

De verkregen dampconcentraties soman kwamen goed overeen met die van de standaardmethode. Het OPTIC-systeem is aldus in staat automatisch en betrouwbaar somandamp te analyseren en is in staat om eventuele bij- en afbraakproducten te registreren.

Toepasbaarheid

Het in dit rapport beschreven systeem lijkt (vooralsnog) bruikbaar voor de automatische analyse van strijdgassen uit de gasgeneratie-opstelling ten behoeve van het testen van detectieapparatuur.

Een geautomatiseerd luchtmonsternamesysteem met een OPTIC gaschromatografie-injector I. Concentratiebepaling van uitstromende soman-damp

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Programmatitel	Projecttitel
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Frequentie van overleg	Projectteam
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Contents

1   Introduction

1.1   Historic perspective

Within the framework of the project 'Evaluation CWA detection equipment' TNO Prins Maurits Laboratory (TNO-PML) performs tests on detectors and monitoring equipment for vapours of chemical warfare agents. This project aims to give scientific support for future chemical detection procurement projects. Within these procurement projects, chemical warfare agent detectors need to be tested with vapours of chemical warfare agents with a known concentration in air, sometimes in the presence of possible interferent gases and vapours. Parameters as response time, residual contamination time and identification of the detected agent are commonly tested.

In order to perform these experiments a vapour generation system is used. This system must be capable of generating a constant concentration of CWA vapour for several hours or even days. In addition the air humidity needs to be controlled within a variation of one percent or less and in some cases even within 0.2 percent.

Our current gas generation system meets these requirements. The system has been described in detail in a TNO-PML document that describes how to operate the system [1]. The concept is based on vapour diffusion of liquid agent contained in a glass vessel. The vapour is then diluted with clean humidified air in a mixing chamber. The dilution can be performed in one or two stages depending on the desired concentration. In the ideal situation the output concentration of CWA vapour is known exactly in real time.

Until about ten years ago we used a Meloy flame photometric analyzer. This instrument can be made specific for either sulfur or phosphorous containing compounds by changing the optical filter. The agent vapour is sucked in the device continuously by its internal pump and the response is measured continuously. Therefore the Meloy acts as a monitor: the signal is plotted in real time continuously. This is a great advantage because one can hook up a detector any time. It is not necessary to wait for the outcome of a chemical analysis. A shortcoming of the Meloy is that it only 'sees' the elements P or S. Any compound containing one or both elements is detected. This means that one can never be certain whether the intact blister or nerve agent is detected or a degradation product or impurity. This uncertainty can only be solved by performing additional chemical analysis. This analysis is usually gas chromatography. Trapping vapour in a solvent or on a solid sorbent followed by GC analysis is already performed by other groups in our laboratory on a routine basis.

When the Meloy flame photometric analyzers could no longer be maintained and repaired because their production was stopped we turned to GC analysis as our standard method to determine the vapour concentration.

Because the vapour concentration is usually very low, vapour has to be preconcentrated on a solid sorbent or in a solvent. In our case we trap the vapour sample on a Tenax sorbent tube. For most agents the tube can then be analyzed by thermodesorption GC analysis. For VX and Lewisite this is not successful. Lewisite decomposes during the thermodesorption at higher temperatures. VX has been shown not to be transferred quantitatively during the thermodesorption process. These agents are therefore removed from Tenax by solvent extraction. The liquid extract is then injected in the GC.

For logistic reasons this analysis has to be performed in a separate laboratory facility at some distance of the actual testing set-up (and by separate analytical personnel). Tenax tubes were collected by us and subsequently handed over to the analysis department. The main drawback of this approach is the delay in concentration

determination. In 1996 we decided to purchase a GC system. This system should also be able to collect and analyze vapour samples automatically and fast. When the run time per sample is short many samples per hour can be obtained. With this approach the concentration can be obtained in near real time with the advantage of chemical analysis. The system will be described in the next chapter. From 1997 to 2003 we used this system. Our experiences led to an adaptation in 2003. The injector part of the system was replaced by the OPTIC GC injector which will be described in detail in this report. Experiments with the OPTIC using the nerve agent soman will be described in detail.

1.2 GC coupled with Sample Preconcentration Trap (SPT)

This system consisted of a Varian 3600 GC equipped with a SPT injector.
The SPT was made up from a sample loop filled with about 75 mg of Tenax sorbent. The sample was collected by adsorption onto a cold trap (usually 20 °C) during a certain time. (Position A in Figure 1). Next the valve was switched to position B and the sample was desorbed by rapid heating. (Position B in Figure 1). The desorbed sample is transferred to the column. Adsorption and desorption time were both 2 minutes in most of our experiments.
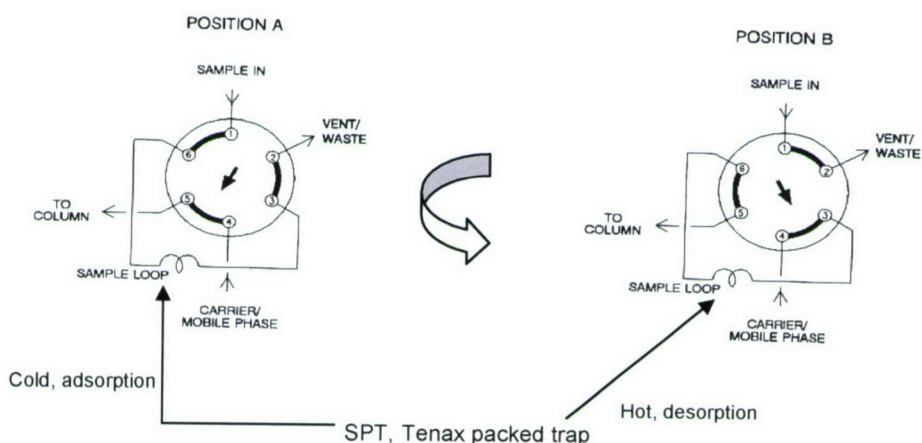

Figure 1   Sampling and injection with 6-port valve including SPT.

The SPT can be heated with a rate of 40 °C/s. Because of this very fast heating rate a cryofocussing step was not necessary, especially not when the GC column temperature was held low during desorption.
The SPT injector worked well for most agents. Most problems were encountered with the nerve agent VX. The effect of humid air on this agent was tremendously.
Besides the problems with VX several drawbacks were encountered.
1   The adsorbent is packed in a metal coiled tube which is assembled in the interior of the GC. One can not see the condition of the adsorbent bed or even if still any adsorbent is in there. Changing the bed by a new one or another material is cumbersome.
2   The desorbed material passes through two ports of the hot sampling valve before reaching the column. This might lead to peak broadening and in humid air to decomposition.

3   The bore of the ports in the valve are rather small. The sampling flow rate is therefore quite low and contact area between vapour and wall quite large.

As mentioned before in 2003 we purchased the OPTIC injector to further optimise the system.

2   OPTIC GC injector as thermodesorption unit

OPTIC 3 is a temperature programmable GC injector with sophisticated temperature and gas flow control. The system can be used for hot split/splitless, cold split/splitless and on-column injections. Facilities such as large volume sampling, thermal desorption and pyrolysis are standard features.

The OPTIC 3 system consists of the injector, the control unit and the control software. The software graphically displays all the system parameters in a real time mode and gives a possibility to save the run-time data for every injection. For our purpose we use it as a thermal desorption unit.

Figure 2 presents a detailed view of the OPTIC injector. The OPTIC is basically a split/splitless injector. The main difference is the temperature control of the OPTIC. The injector can be heated with a maximum rate of 16 °C per second. Therefore the injector is mainly used for large volume injection. When a large volume of solvent is injected at low temperature of the injector equipped with a suitable liner the solute is trapped in the liner while the solvent is vented through the open split line. When the solvent is sufficiently removed the split line closes and the injector is rapidly heated to inject the solutes.
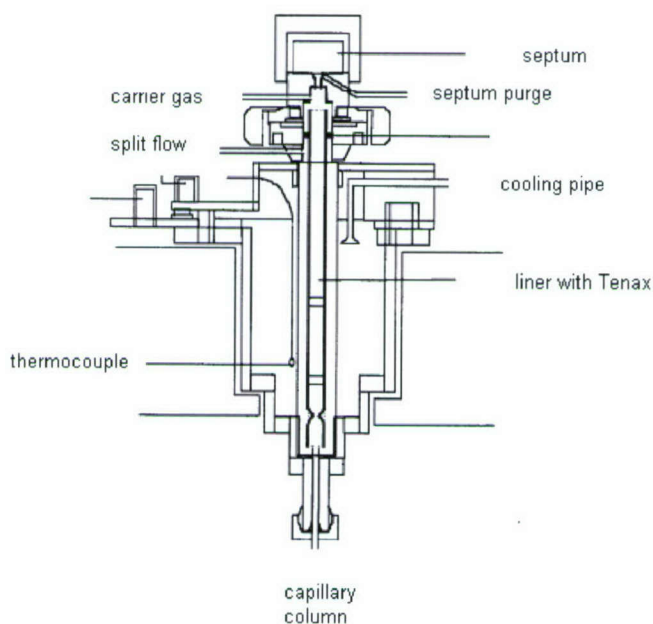

Figure 2   Cross sectional view of the OPTIC GC injector. The cooling pipe can be connected to air,
                liquid CO_2 or liquid nitrogen.

In our application the OPTIC injector is configured as a thermal desorber by replacing the liner with a glass tube packed with Tenax adsorbent. The injector is placed in an air sampling system which can take air samples from the vapour generation system automatically. The whole system is depicted in Figure 3.

Not shown in the picture is the control box for the OPTIC and the personal computer. The PC contains the ATAS Evolution software to control the OPTIC and STAR software to control the Varian 3600GC. The six port valve V2 has two positions either sampling or injection:

Sampling (Figure 3a)

In this position the sample flows trough the sample inlet line from port 4 to port 5 and then via the T-connector upwards through the Tenax tube. At the top of the injector the gas flows away via port 2 and 3 to the vacuum line. The flow rate is controlled by the mass flow controller in this line. The carrier gas line is closed by a solenoid valve which prevents the intake of carrier gas instead of sample air.

Injection (Figure 3b)

In this position the sample is bypassed directly to the vacuum line from port 4 to port 3. The carrier gas is flowing through the Tenax tube directly into the column. To prevent the carrier gas from flowing to the side arm of the T-connector a silica restrictor is connected via port 5 and 6. The same type restrictor is placed on port 1 to prevent the carrier gas from flowing to this sampling line via port 2.
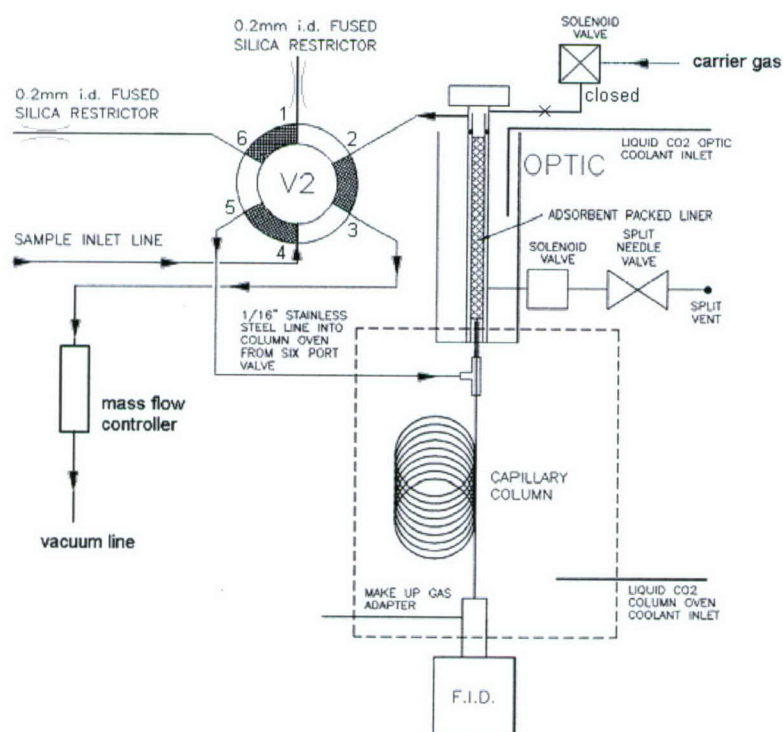

Figure 3a   Flow path of air sampling system with OPTIC thermodesorption injector in the sampling position.
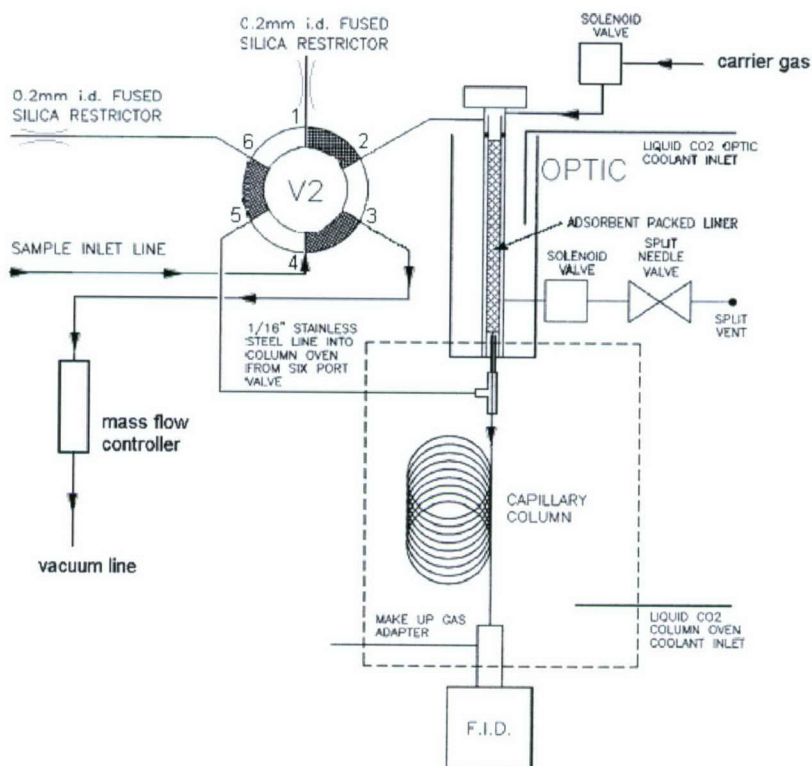
Figure 3b   Flow path of air sampling system with OPTIC thermodesorption injector in the inject position.

As mentioned before the air sampling system is automated. The sequence of events is as follows:

1   GC run is started, OPTIC waits in ready in mode;
2   OPTIC is started by relays in GC;
3   6 port valve is switched in sampling position. Vapour is trapped on Tenax tube at around 25 °C during a preset period, usually 2 minutes;
4   the six port valve is switched to the inject position and heated with 16 °C/s. Vapour is desorbed onto the GC column, which is held at low temperature to focus the vapour. Desorption time is usually 2 minutes;
5   after about 1 minute the temperature program of the column is started;
6   after elution of the component peak the OPTIC cools down to 25 °C again;
7   when the GC run is complete the oven cools down and when stable the next run is started.

Figure 4 gives an impression of the way the OPTIC is programmed. The graph presents the programmed parameters from the OPTIC which are stored in a method file. During a run the actual values are also displayed in the same type of graph. One should keep in mind that the OPTIC itself is started by the Gas Chromatograph. Each run is started by the sample list in the Star software from the GC.
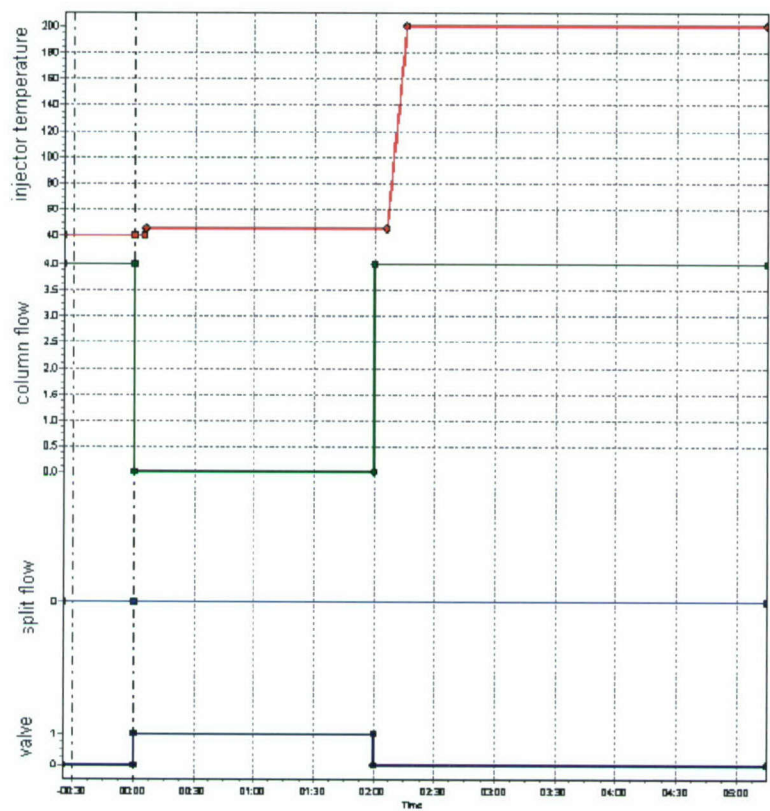
Figure 4   Graphical presentation of programming the OPTIC. During sampling the six port valve is in
               position 1 (dark blue curve), the injector temperature (red curve) is low and the column flow
               (green curve) is 0. After 2 minutes adsorption, the trap is desorbed by rapid heating to 200 °C.
               At the same time the column flow is started and the desorbed components are injected onto
               the GC column. The split flow (light blue curve) is not used in this example (splitless
               injection mode).

3   Description of experiments
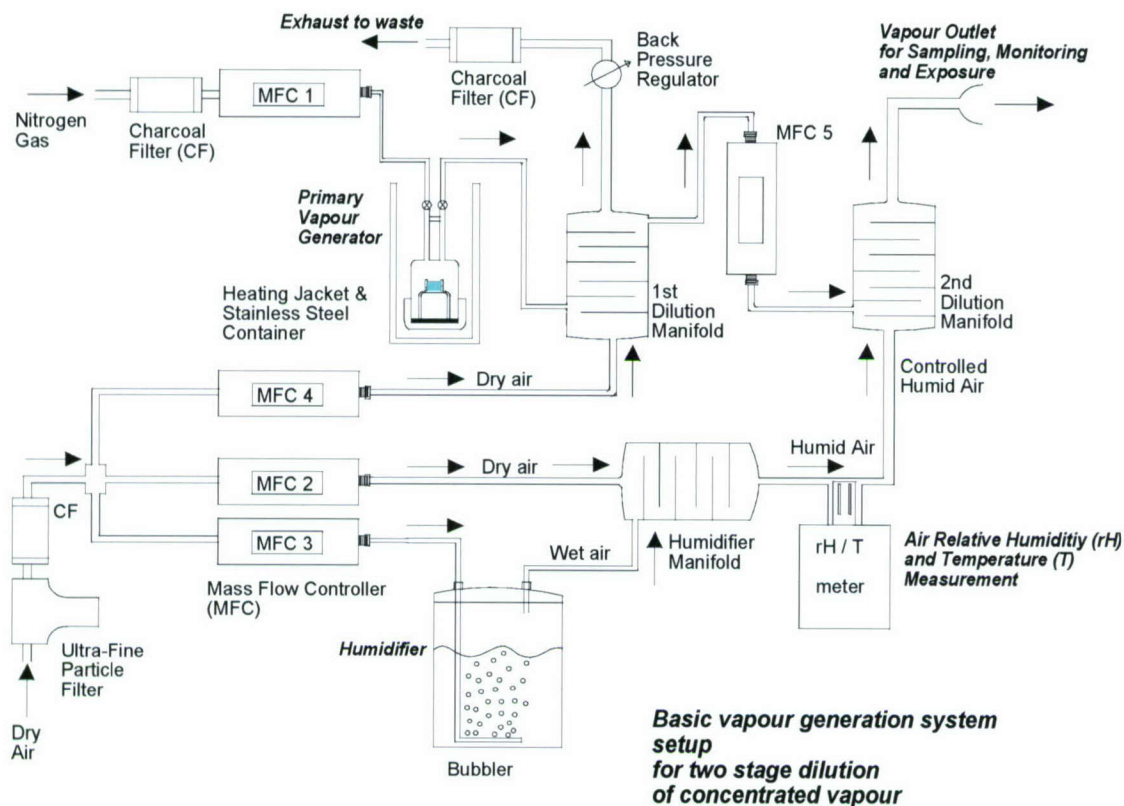
Figure 5   Lay out of PML gas/vapour generation system.

3.1   Generation of soman vapour

To test and validate the OPTIC injector, the OPTIC was allowed to sample chemical warfare agent vapour from the vapour generation system. In Figure 5 the vapour generation system is depicted.

The 'Fortuin' primary generation vessel was equipped with a large area evaporation tube.

The diameter was 2.8 cm and height 11 mm containing 2 ml of liquid soman. The generation system was configured as a two stage dilution set up. The flow rates were controlled with mass flow controllers (Bronkhorst High Tec). The generation was started with the following values:

- $F1 = 200$ ml/min, primary nitrogen stream;
- $F2 = 15$ l/min, dry air;
- $F3 = 0$ l/min, humidified air;
- $F4 = 3.75$ l/min;
- $F5 = 0.5$ l/min;
- $F6 = 80$ ml/min, sampling flow rate to OPTIC.

The humidity was measured with a Novasina MIK 3000-C capacitive RH meter.
The generation vessel was kept at ambient temperature.
The sample inlet line was a 2 meter piece of 1/8 inch Teflon tubing which was allowed
to sample from the exhaust of the generation system.

3.2 Calibration of Flame ionisation detector (FID)

For this purpose the column was detached from the OPTIC and attached to the Varian
Septum equipped Programmable Injector (SPI). This injector can be temperature
programmed. A stock solution of 10 mg soman in 50 ml hexane was made (200 ng/μl).
Several dilutions in hexane were made in the concentration range from 2 to 200 ng/μl.
A volume of 1 μl was injected with a Hamilton 1 μl plunger in needle syringe. At least
three injections were done at each concentration. In most cases two peaks from both
stereoisomers were obtained. The peak areas from both peaks were added yielding a
peak area which was much the same as the peak area from a single peak, as sometimes
obtained.

GC Conditions: SPI injector: 0.5 min at 50 °C, 20 °C/min to 200 °C
Column: 8 mCPsil5CB, Df=1 μ.
Oven: 1 min 50 °C, 20 °C/min to 200 °C.

3.3 Quantification of soman vapour with OPTIC

The effects of several parameters on the performance of the OPTIC system were
investigated. The system was also calibrated against an off line sampling method which
employs solvent desorption of Tenax tubes.

3.3.1 *Effect of humidity*
Only two air humidities were used for this test: dry air which has a humidity below 1%
and a humidity of 50% which was obtained by setting $f2 = f3 = 7.5$ l/min. At both
humidities samples were taken by OPTIC for several hours. The sampling flow rate was
25 ml/min and the sampling time was 2 minutes. The cycle time per sample, depending
mainly on the retention time of the soman peak, was 12 minutes.

3.3.2 *Effect of trapflow rate*
This is a parameter that has to be optimized. On one hand the flow rate should be as
high as possible to trap much vapour in a short time. On the other hand breakthrough of
the sorbent bed can occur. The maximum flow rate might also be limited by the highest
possible pressure drop across the bed. Since the sampling mass flow controller is
connected to vacuum, the maximum pressure drop is always less then 1 ato.
The set flow rates were: 40, 80, 120 and 160 ml/min. The sampling time was 2 minutes.
At each flow rate samples were taken for several hours. The cycle time was again
12 minutes.

3.3.3 *Comparison with off-line sampling and analysis*
Many parameters are involved in the concentration determination with the OPTIC like
sampling flow rate, sampling time, desorption time, desorption temperature, length of
sample inlet line, kind of tubing etc. Some will be more critical then others. Some are
known from experience with thermodesorption analysis. The desorption temperature for
instance is not critical, commonly a value of 200 °C is chosen.

Since it's not possible to investigate all parameters, a comparison with another analysis method was performed.

The off-line method is called this way because the sample tube is not attached in line of the sampling GC system. Instead it is removed manually from the vapour outlet and desorbed manually with solvent. Solvent desorption has proven to be a reliable straightforward method. Hexane is used because it matches the apolarity of the Tenax adsorbent. Therefore the wetting of Tenax by hexane is good which can be observed by a slight swelling of the sorbent.

Chrompack glass adsorption tubes filled with 100 mg *Tenax* TA 20-35 Mesh were used. A *Bronkhorst* mass flow controller connected to the vacuum line at the outlet side and to the generation system (atmospheric pressure) at the inlet side controlled the sampling flow rate. The flow rate was adjusted to 0.2 L/min. The inlet side of the tube was held in the outlet of the vapour generation system for a period of minutes to hours depending on the concentration. After sampling, the tube was detached from the generation system and the adsorbed vapour was desorbed from the *Tenax* with hexane (*Lichrosolv* from *Merck*, Germany). The effluent from the tube was collected in a 4-ml screw neck glass vial. The volume of collected hexane effluent was determined by the weight (density hexane is 0.66 g/ml). In all cases the volume of the effluent in each fraction was approximately 1 ml. Two fractions were collected which appeared to be sufficient for complete desorption. 1 μl of the collected hexane solution was injected in the Varian 3600GC equipped with SPI injector and FID.

4   Results

4.1   Calibration of Flame ionisation detector

The results of the calibration of Flame ionisation detector are presented in Figure 6 in a log-log plot. The equation of the linear curve fit is:

log Peak area = 3.119 + 1.053 log (ng GD)
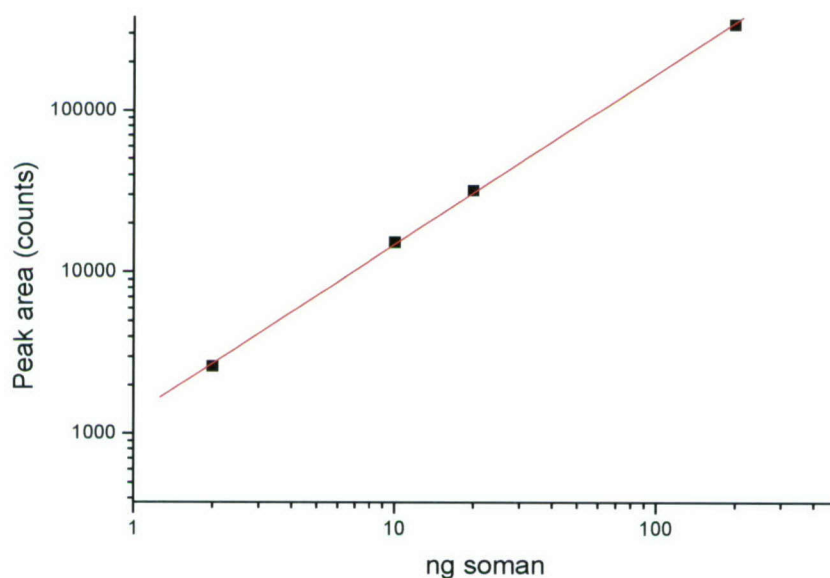
Figure 6   Calibration of FID on soman. 1 µl solutions of GD in hexane were injected.

The use of this calibration curve is not accurate enough in the low concentration range. For this purpose the high 200 ng point is skipped and the point 0,0 is included which is essentially a blank chromatogram. This point cannot be included in the logarithmic plot. The equation of the linear curve fit of this graph (Figure 6) is:

Peak Area = -377 + 1608 (ng GD)
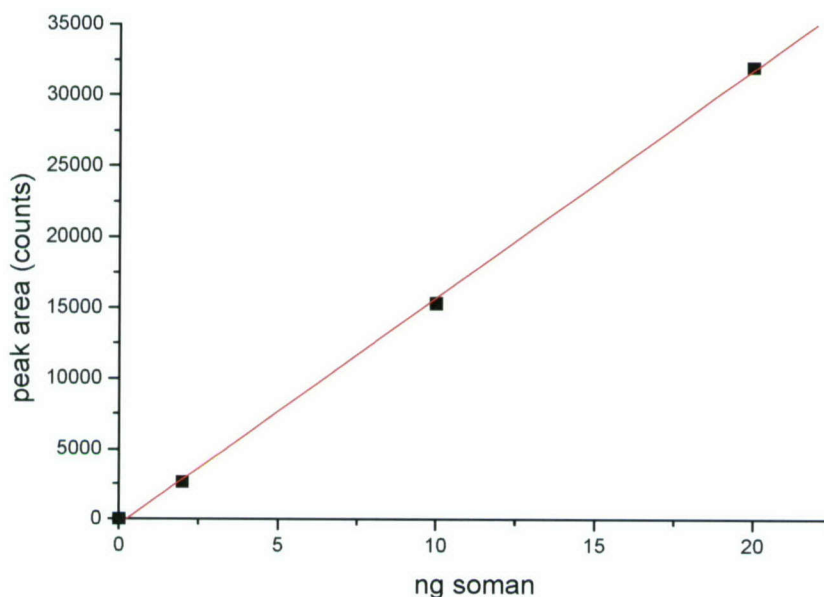
Figure 7   Calibration of FID on soman in the low concentration range.

4.2   Quantification of soman vapour

4.2.1   Effect of humidity

Table 1   Influence of air humidity on the concentration soman. The sampling volume was 50 ml.

RH (%)	Peak area (counts)	Number of measurements, n	Standard deviation, σ	Concentration GD (mg/m³)
< 1	83790	7	7%	1.05
49	83332	5	2%	1.04

It's clear that, in this range, humidity does not effect the concentration determined by OPTIC. It remains very interesting what the effect will be at high humidity, say 90% or even higher.

4.2.2   Effect of trap flow rate

The actual trap flow rate was measured with a soap bubble meter at the sample inlet line. It appeared to be much less then the set flow rate. It was found that besides air from the sample inlet line, carrier gas was sucked in as well. For this reason a solenoid valve was mounted in the carrier gas supply line. This valve closes the carrier gas supply line during sampling.

The amount of soman increases linear with trap flow rate and thus with trap volume since the trap is 2 minutes at each flow rate. Therefore the calculated vapour concentration is independent of trap flow rate in this range. Since the flow rates are rather small due to the aforementioned leakage problems, higher flow rates still have to be investigated.
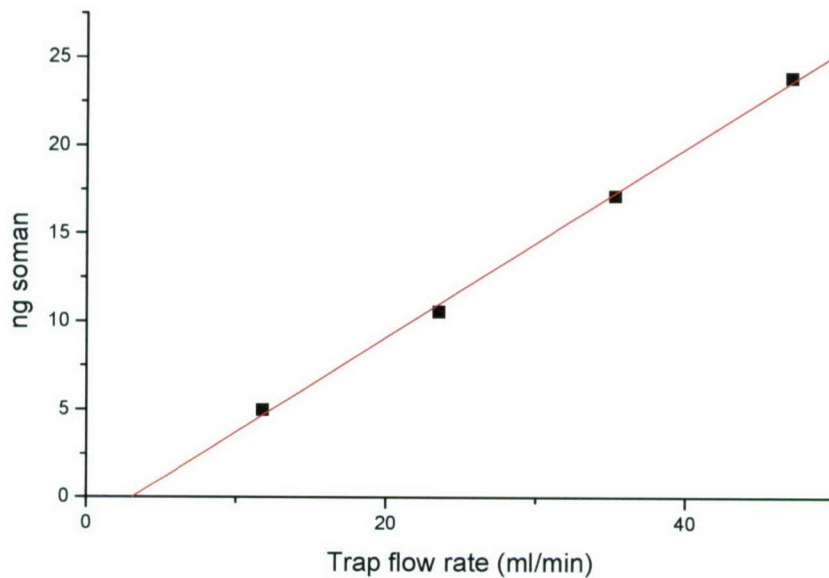
Figure 8   Influence of trap flow on the amount of recovered soman. Trap time is 2 minutes. Each point
            in the graph is the average of 5 data points with a standard deviation of 1 to 5%.

4.2.3   *Comparison with off-line sampling and analysis*

In the text below an example is given of how the concentration of soman vapour is
calculated in case of off and on-line sampling. Note that for off-line sampling (solvent
extraction) the sampling time is very long as compared to thermodesorption in order to
inject a comparable amount into the GC. This is of course due to the fact that only 1 μl
out of 0.69 ml solvent is injected where in the case of thermodesorption all trapped
vapour is injected.

Off-line

The trapflow was 90% x 200 mln/min = 180 mln/min. The vapour was trapped for
3 hours and 24 minutes. The avarage peak area of a 1 μl injection was 153000 counts.
According to calibration plot in Figure 5 this yields 91.5 ng soman in 1 μl. The amount
of hexane was 0.69 ml. The amount of desorbed soman is then:

0.69 ml x 91.5 μg/ml = 63.1 μg.

The calculated concentration soman is then:

$C_{GD,offline}$ *= 63.1 μg / (204 min x .18 l/min) = 1.7 μg/l = 1.7 mg/m³.*

On-line

The average peak area was 132000 counts. This yields 79.6 ng soman. The vapour was
trapped for 2 minutes at 25 ml/min.
The calculated concentration soman is then:

$C_{GD, online}$ *= 79.6 ng / 50 ml = 1.6 ng/ml = 1.6 mg/m³.*

Table 2   Comparison between solvent extraction and OPTIC thermodesorption of the determined concentration soman vapour.

RH (%)	Off-line, solvent desorption	Amount trapped soman, off-line	On-line, OPTIC	Amount trapped soman, on-line
< 1	1.7 mg/m^3	63 µg	1.6 mg/m^3	79 ng
< 1	Not measured		0.035 mg/m^3	1.7 ng
90	0.049 mg/m^3	1.2 µg	0.038 mg/m^3	1.9 ng
90	0.041 mg/m^3	1.6 µg	0.037 mg/m^3	1.9 ng

The concentrations soman vapour as determined by the OPTIC thermodesorption system are in excellent agreement with the solvent desorption method.

The lower concentrations are not far from the detection limit of the FID. When still lower concentrations have to be determined the sampling time must increase and/or a more sensitive detector must be used.

5   Conclusions and discussion

The automated air sampling and analysis system built around the OPTIC GC injector was successfully employed in our laboratory for the unattended near real time monitoring and analysis of soman vapour. The system provides a semi continuous concentration level of soman in dry and humid air.

With respect to the previously employed system, described in Section 1.2, several substantial improvements were reached with the current system. The OPTIC GC injector, through its design, has markedly better analytical performance, i.e. it allows better quantification of agent concentration and it is clearly more flexible in allowing rapid experimental adjustments to be carried out.

Naturally other chemical warfare agents have to be tested. No problems are to be expected with sarin and sulfur mustard. We expect that Lewisite, which is very reactive, cannot be thermodesorbed at all. This agent probably has to be solvent desorbed and derivatised to a GC compatible agent.

The low volatile VX is expected to be much more difficult than the other nerve agents because it adsorbs on all kinds of materials. Specially deactivated tubing or heated sample transfer lines might be a solution. The next report in this series will deal with VX only. A lot of effort has been made and will be spent especially for the analysis of VX because this agent has a lot of attention in the world.

The, presented in this report, automated vapour generation monitor allows, in combination with the chemical warfare agent vapour generation system, rapid and accurate testing of chemical warfare agent detectors. With an optimised system more different concentrations can be tested at accurately determined concentrations. This makes more extensive testing for chemical warfare agent detector procurement programmes possible. A second benefit is that the monitoring approach implemented in this report for the laboratory chemical warfare agent vapour generation system is a valid approach for the development of chemical warfare agent field monitoring. This report therefore also allows a better judgement of possible future chemical warfare agent field monitors employing this approach.

6   References

[1]   Olivier, R.C.M.,
      Het gebruik van het Damp-/Gasgeneratiesysteem,
      (Use of the vapour- and/or gas generation system),
      Q214-W-116, 2 september 2003.

7 Signature

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15. ABSTRACT (MAXIMUM 200 WORDS (1044 BYTE))

An automated air sampling and analysis system was built around the OPTIC GC injector. The system was successfully employed for the unattended near real time monitoring and analysis of soman vapour. With respect to the previously employed system several substantial improvements were reached with the current system. The OPTIC GC injector, through its design, has markedly better analytical performance, i.e. it allows better quantification of agent concentration and it is clearly more flexible in allowing rapid experimental adjustments to be carried out.
The OPTIC automated vapour generation monitor allows, in combination with the chemical warfare agent vapour generation system, rapid and accurate testing of chemical warfare agent detectors. With the OPTIC system more different concentrations can be tested at accurately determined concentrations. This makes more extensive testing for chemical warfare agent detector procurement programmes possible.

16. DESCRIPTORS   IDENTIFIERS

Vapour generation, Automated quantification, Soman,
OPTIC injector

17a. SECURITY CLASSIFICATION (OF REPORT)	17b. SECURITY CLASSIFICATION (OF PAGE)	17c. SECURITY CLASSIFICATION (OF ABSTRACT)
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18. DISTRIBUTION AVAILABILITY STATEMENT	17d. SECURITY CLASSIFICATION (OF TITLES
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